

§ 162.050-39 Measurement of oil content.

(a) *Scope.* This section describes the method and apparatus to be used in measuring the oil content of a sample taken in approval testing of each separator, monitor, or alarm. Light oil fractions in the sample, with the exception of volatile components lost during extractions, are included in each measurement.

(b) *Summary of method.* Each sample is acidified to a low pH and extracted with two volumes of solvent. The oil content of the sample is determined by comparison of the infrared absorbance of the sample extract against the absorbance of known concentrations of a reference oil in solvent.

(c) *Apparatus.* The following apparatus is used in each measurement:

(1) Separatory funnel that is 1000 ml. or more in volume and that has a Teflon stopcock.

(2) Infrared spectrophotometer.

(3) A cell of 5 mm. pathlength that has sodium chloride or infrared grade quartz with a minimum of 80 percent transmittance at 2930 cm^{-1} . (This cell should be used if the oil content of the sample to be measured is expected to have a concentration of between 2 p.p.m. and 80 p.p.m.)

(4) A cell of pathlength longer than 5 mm. that has sodium chloride or infrared grade quartz with a minimum of 80 percent transmittance at 2930 cm^{-1} . (This cell should be used if the oil content of the sample to be measured is expected to have a concentration of between 0.1 p.p.m. and 2 p.p.m.)

(5) Medium grade filter paper.

(6) 100 ml. glass stoppered volumetric flasks.

(d) *Reagents.* The following reagents are used in each measurement:

(1) Hydrochloric acid prepared by mixing equal amounts of concentrated, reagent grade hydrochloric acid and distilled water.

(2) Reagent grade sodium chloride.

(3) One of the following solvents:

(i) Spectrographic grade carbon tetrachloride.

(ii) Reagent grade Freon 113, except that this solvent may not be used to analyze samples in approval testing of cargo monitors. (Ucon 113, Genatron

113, or an equivalent fluorocarbon solvent are also acceptable.)

(4) Reference oil, which is the oil used in the portion of the test during which the sample is collected.

(5) Stock reference standard prepared by weighing 0.30 g. of reference oil in a tared 100 ml. volumetric flask and diluting to 100 ml. volume with solvent.

(e) *Preparation of calibration standards.* A series of dilutions is prepared by pipetting volumes of stock reference standard into 100 ml. volumetric flasks and diluting to volume with solvent. A convenient series of volumes of the stock reference standard is 5, 10, 15, 20, and 25 ml. The exact concentrations of the dilutions in milligrams of oil per 100 milliliters of diluted stock reference standard are calculated. The calibration standards are the dilutions.

(f) *Extraction.* (1) A reagent blank is carried through each step described in this paragraph and paragraph (g) of this section.

(2) The pH of each sample is checked by dipping a glass rod into the sample and touching the rod with pH-sensitive paper to ensure that the pH is 2 or lower. More acid is added if necessary until the pH is 2 or lower. The glass rod is then rinsed in the sample bottle with solvent.

(3) The sample is poured into a separatory funnel and 5 g. of sodium chloride are added.

(4) Fifty (50) ml. of solvent are added to the sample bottle. The bottle is capped tightly and shaken thoroughly to rinse its inside. The contents of the bottle are then transferred to the separatory funnel containing the sample and extracted by shaking vigorously for 2 minutes. The layers are allowed to separate.

(5) The solvent layer is drained through a funnel containing solvent moistened filter paper into a 100 ml. volumetric flask.

(6) Fifty (50) ml. of solvent are added to the sample bottle. The bottle is capped tightly and shaken thoroughly to rinse its inside surface. The contents of the bottle are then transferred to the separatory funnel containing the water layer of the sample. The contents of the separatory funnel are then extracted by shaking vigorously for 2

minutes. The layers are allowed to separate. The solvent layer is then drained through a funnel containing solvent moistened filter paper into the volumetric flask containing the solvent layer of the sample.

(7) The tips of the separatory funnel, filter paper, and funnel are rinsed with small portions of solvent and the rinsings are collected in the volumetric flask containing the solvent layer of the sample. The volume is adjusted with solvent up to 100 ml. The flask is then stoppered and its contents are thoroughly mixed.

(8) The water layer remaining in the separatory funnel is drained into a 1000 ml. graduated cylinder and the water volume estimated to the nearest 5 ml.

(g) *Infrared spectroscopy.* (1) The infrared spectrophotometer is prepared according to manufacturer instructions.

(2) A cell is rinsed with two volumes of the solvent layer contained in the volumetric flask. The cell is then completely filled with the solvent layer. A matched cell containing solvent is placed in the reference beam.

(3) If a scanning spectrophotometer is used, the solvent layer in the cell and

the calibration standards are scanned from 3200 cm^{-1} to 2700 cm^{-1} . If a single beam or non-scanning spectrophotometer is used, the manufacturer's instructions are followed and the absorbance is measured at or near 2930 cm^{-1} .

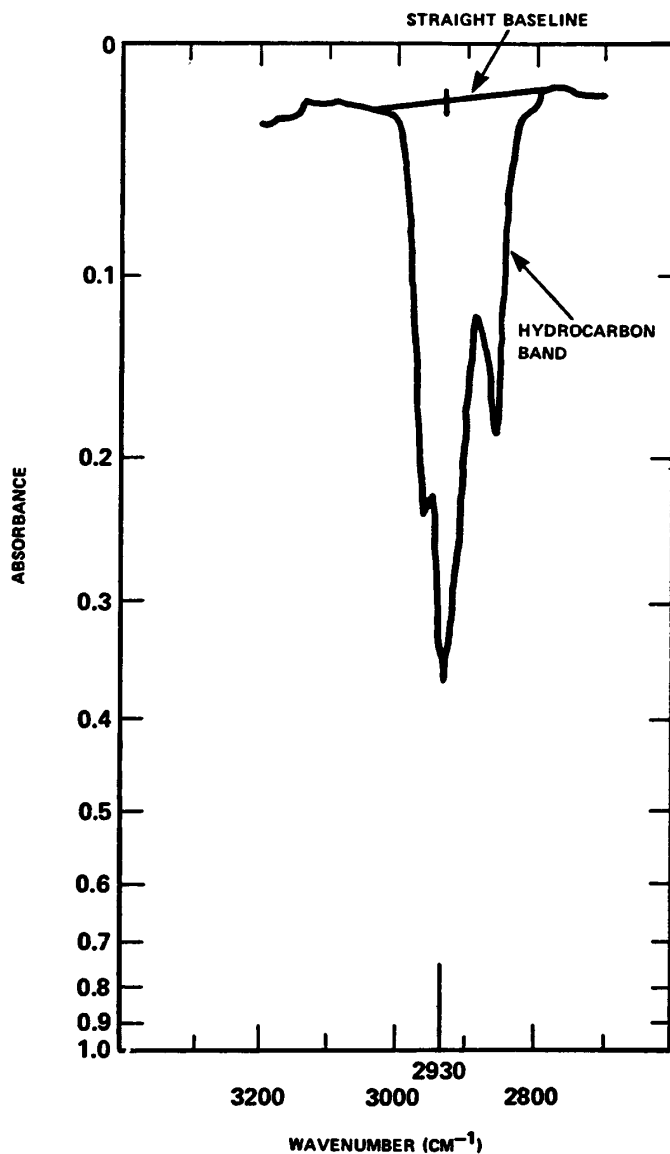
(4) If the scan is recorded on absorbance paper, a straight baseline of the type described in Figure 162.050-39(g) is constructed. To obtain the net absorbance, the absorbance of the baseline at 2930 cm^{-1} is subtracted from the absorbance of the maximum peak on the curve at 2930 cm^{-1} .

(5) If the scan is recorded on transmittance paper, a straight baseline is constructed on the hydrocarbon band plotted on the paper. The net absorbance is:

$$\log_{10} \frac{\%T(\text{baseline})}{\%T(\text{peak maximum})}$$

(6) A plot is prepared for net absorbance vs. oil content of the calibration standards or of the percentages of stock reference standard contained in the calibration standards.

FIGURE 162,050-39(g) - SPECTRUM ILLUSTRATING BASELINE CONSTRUCTION



(7) If the net absorbance of a sample determined by the calibration plot exceeds 0.8 or the linear range of the spectrophotometer, a dilution of the solvent layer contained in the volumetric flask after completing the step

described in paragraph (f)(7) of this section is prepared by the pipetting an appropriate volume of the solvent layer into a second volumetric flask and diluting to volume with solvent. If the net absorbance is less than 0.1 when determined in accordance with the procedures in this paragraph, it is recalculated using a longer pathlength cell.

(h) *Calculations.* (1) The plot described in paragraph (g)(6) of this section is used to determine the milligrams of oil in each 100 ml. of solvent layer contained in the volumetric flask after completing the steps described in paragraph (f) or paragraph (g)(7) of this section.

(2) The oil content of the sample is calculated using the following formula:

oil content of sample = $R \times D \times 1000 / V$

R = mg. of oil in 100 ml. of solvent layer determined from plot.

D = 1 or, if the step described in paragraph (g)(7) of this section is performed, the ratio of the volume of the second volumetric flask described in that paragraph to the volume of solvent layer pipetted into the second volumetric flask.

V = The volume of water in milliliters drained into the graduated cylinder at the step described in paragraph (f)(8) of this section.

(3) The results are reported to two significant figures for oil contents below 100 mg/l and to three significant figures for oil contents above 100 mg/l. The results are converted to p.p.m.

PART 163—CONSTRUCTION

Subpart 163.001 [Reserved]

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SOURCE: CGFR 50-30, 16 FR 1086, Feb. 6, 1951, unless otherwise noted.

Subpart 163.001 [Reserved]

Subpart 163.002—Pilot Hoist

SOURCE: CGD 74-140, 46 FR 63287, Dec. 31, 1981, unless otherwise noted.

§ 163.002-1 Scope.

(a) This subpart contains standards and approval and production tests for pilot hoists used on merchant vessels.

(b) The requirements in this subpart apply to a pilot hoist designed for use along a vertical portion of a vessel's hull.

§ 163.002-3 Applicable technical regulations.

(a) This subpart makes reference to the following Coast Guard regulations in this chapter:

(1) Subpart 58.30 (Fluid Power and Control Systems).

(2) Section 94.33-10 (Description of Fleet Angle).

(3) Part 111 (Electrical System, General Requirements).

(4) Subpart 163.003 (Pilot Ladder).

(b) [Reserved]

§ 163.002-5 Definitions.

(a) *Maximum persons capacity* means—

(1) If the hoist has a rigid ladder, one person; or

(2) If the hoist has a platform, one person per square meter (10.75 sq. ft.) or fraction thereof of platform area (including hatch area);

(b) *Working load* means the sum of the weights of—